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05FEB01 E603175-1 D02877
P01/7700 0.00-0102736.6

1.	Your reference	CRO 50925/GB
2.	Patent application number *(The Patent Office will fill in this part)*	0102736.6 — 5 FEB 2001
3.	Full name, address and postcode of the or of each applicant *(underline all surnames)*	CROSFIELD LIMITED Bank Quay Warrington CHESHIRE WA5 1AB United Kingdom
	Patents ADP number *(if you know it)*	6449610…
	If the applicant is a corporate body, give the country/state of its incorporation	United Kingdom
4.	Title of the invention	DISINTEGRANTS AND A PROCESS FOR THEIR MANUFACTURE
5.	Full name of your agent *(if you have one)*	JACKSON, John Derek
	"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	Crosfield Limited Intellectual Property Department Bank Quay Warrington Cheshire WA5 1AB
	Patents ADP number *(if you know it)*	7645250…
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11. I/We request the grant of a patent on the basis of this application

Signature John Jock Jackson Date 05 February 2001
AGENT FOR THE APPLICANTS

12. Name and daytime telephone number of person to contact in the United Kingdom — Kath Chester 01925 416243

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CRO 50925

DISINTEGRANTS AND A PROCESS FOR THEIR MANUFACTURE

This invention relates to disintegrants useful in solid dosage forms and, in particular, in compositions intended to be formed into moulded bodies for use in, for example, home care products, personal care products, food, agricultural, industrial and pharmaceutical applications.

One particular use for disintegrants is in detergent compositions in the form of tablets which have been widely described and are currently enjoying increasing popularity with consumers. They are described, for example, in GB 0 911 204 (Unilever), US 3 953 350 (Kao), DE 19 637 606 (Henkel), EP 0 711 827 (Unilever) and WO 98/40463 (Henkel). Tablets for machine dishwashing applications are described in, for example, WO 96/28530 (P&G). Tabletted detergents and cleaning compositions have several advantages over powdered or liquid compositions: they are easier to dispense and handle, do not require measuring to obtain the correct dose and, being compact, are more economical to store and transport.

Tablets of cleaning compositions are generally made by compression or compaction of a quantity of a composition in the form of particles. Production of tablets which are sufficiently hard and strong to withstand storage and handling requires a relatively high pressure to be used in this compaction process. It is then necessary that, despite this compaction, the tablets are able to disperse and dissolve rapidly when added to wash water.

One approach to achieving good dispersion of the tablet is to include in the tablet a particulate insoluble, but water-swellable, agent. These particles then swell with ingress of water, leading to stresses in the tablet and thence to break-up of the tablet. Thus WO 98/55583 (Unilever) describes the use of 'water-insoluble, water-swellable polymeric material' which 'promotes disintegration of the tablets in water'. Typical swelling agents which have been disclosed as possible tablet disintegrating agents are starches, cellulose and cellulose derivatives, alginates, dextrans, cross-linked polyvinyl pyrrolidones, gelatines and formaldehyde casein as well as a wide variety of clay minerals and certain ion-exchange resins.

These water-swellable agents have no function in fabric washing except to aid tablet disintegration. Furthermore, because they are insoluble and of relatively large particle size, they tend to deposit on clothes during the wash (see, for example, WO 98/55575 (Henkel) and WO 98/55582) As a result, several attempts have been made to minimise the deposition of these disintegrants, for example by combining such a water-swellable, insoluble disintegrant with a second, highly soluble disintegrating aid – see WO 98/55582 (Unilever). Other attempts have included use of a preferred particle size of the disintegrant. Thus, for example, WO 98/55583 (Unilever) claims use of such material at a particle dimension of at least 400 μm to give more efficient disintegration. On the other hand, WO 98/55575 (Henkel) teaches the use of cellulose disintegrating aids with a particle size of less than 100 μm, in order to minimise deposition. This material is co-granulated with 'microcrystalline cellulose and/or one or several ingredients of detergents and cleaning agents'.

Despite these attempts to provide satisfactory tablet disintegration with minimal deposition, there is still a need for an improved composition, particularly for one that retains good dispersion properties on storage.

The process and the disintegrant of this invention can be used in a number of applications where tablets are used and, in particular, it has been surprisingly found that a detergent tablet which disintegrates readily and retains its easy disintegration on storage can be produced by incorporating into the composition used to produce the tablet a co-granule according to an aspect of this invention.

According to one aspect of the invention a process for the preparation of a disintegrant, suitable for use in a composition in the form of a moulded body, comprises forming by a dry granulation process a granular composition comprising a swelling clay and a water insoluble inorganic material.

According to a preferred embodiment of the invention a process for the preparation of a disintegrant, suitable for use in a composition in the form of a moulded body, comprises forming by a dry granulation process a granular composition comprising a swelling clay, a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent of the combined weight of said swelling clay, said water insoluble material and said water-swellable agent.

Also according to the present invention there is provided a composition suitable for use as a disintegrant in a composition in the form of a moulded body, said composition being in the form of granules comprising a swelling clay, a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent of the combined weight of said swelling clay, said water insoluble inorganic material and said water-swellable agent.

We have found that if a water-swellable agent is co-granulated with a swelling clay and a water insoluble inorganic material before being incorporated into a moulded body, particularly in the form of a tablet, then the water-swellable agent is much more efficient in aiding disintegration of the body in use than when it is not co-granulated. Thus, less of the water-swellable agent needs to be added for effective disintegration. When the disintegrant is used in tablets for cleaning materials this effect lowers the probability of deposition of the water-swellable agent on the substrate being cleaned. Moreover, tablets prepared using the granulates according to this invention disintegrate in use readily even after being stored for some time.

The present invention seeks to provide a disintegrant which is useful in a particulate composition in the form of a moulded body and which is relatively inexpensive to manufacture and sufficiently robust to withstand handling during production and packaging processes but readily breaks up and dissolves when contacted with an aqueous medium during the process for which it is intended.

When present, the water-swellable agent preferably comprises, in its anhydrous state, no more than 15 per cent, more preferably no more than 10 per cent, of the combined weight of said swelling clay, said water insoluble inorganic material and said agent in the granular disintegrant. In a typical preferred composition of the invention, the water-swellable agent comprises, in its anhydrous state, no more than 8 per cent, e.g. 7.5 per cent or less, of the combined weight of said swelling clay, said water insoluble inorganic material and said agent. Generally, at least 1 per cent of the combined weight of the water-swellable agent, swelling clay and the water insoluble inorganic material in the granules comprises water-swellable agent.

A feature of the preferred aspect of this invention is the relatively small amount of water-swellable agent that may be employed while securing satisfactory properties for a compacted composition, particularly a cleaning composition. Frequently, the amount of water-swellable agent in a cleaning composition containing the disintegrants of the invention is less than 2 per cent by weight. Preferably, the amount is less than 1 per cent by weight of a cleaning composition but usually at least 0.2 per cent by weight of the water-swellable agent is present in a cleaning composition.

A number of clays are known as swelling clays and particularly suitable swelling clays for use in this invention include smectite clays. The term "smectite" is used to classify geologically certain three-layer swelling clays. The term "swelling" as used herein relates to the ability of the layered clay structure to swell, or expand, on contact with water. Clays having a smaller particle size are preferred in detergent applications since they give fewer problems with residues on a substrate that has been washed.

There are two distinct classes of smectite clays. In the first, aluminium oxide is present in the silicate crystal lattice; in the second class, magnesium oxide is present in the silicate crystal lattice. The general formulas of these smectites are $Al_2(Si_2O_5)_2(OH)_2$ and $Mg_3(Si_2O_5)(OH)_2$ for the aluminium and magnesium oxide type clay, respectively. The range of the water of hydration can vary with the processing to which the clay has been subjected. Such processing does not significantly affect the swelling characteristics of the hydrated clays as this is dictated by the silicate lattice structure. Furthermore, atom substitution by iron and magnesium can occur within the crystal lattice of the smectites, while metal cations such as Na^+, Ca^{2+}, as well as H^+, can be co-present in the water of hydration to provide electrical neutrality.

The three-layer, swelling aluminosilicate clays useful herein are further characterised by a dioctahedral crystal lattice, while the swelling three-layer magnesium silicate clays have a trioctahedral crystal lattice.

The clays employed in the invention can contain cationic counterions such as protons, sodium ions, potassium ions, calcium ions and magnesium ions. It is customary to distinguish between clays on the basis of one cation predominantly or exclusively absorbed. For example, a sodium clay is one in which the absorbed cation is predominantly sodium. Such

absorbed cations can become involved in exchange reactions with cations present in aqueous solutions.

The smectite clays used in the present invention are commercially available. Such clays include, for example, montmorillonite, hectorite, saponite volchonskoite, nontronite and sauconite. Such clays are available under commercial names such as Bentonite L, H and MB, all from SCP, USA, and the trade name VOLCLAY from American Colloid Co. Some commercially available clays are produced by treating naturally-occurring clays, for example, by treating calcium bentonite (a form of montmorillonite) with a compound of sodium and/or with other cations. A preferred bentonite clay for use in this invention is a calcium bentonite clay which has been treated with sodium carbonate and which is sold under the trade names "Brebent" and "Fulgel" by Laporte plc. Other suitable clays are available commercially. The smectite minerals obtained under these commercial and trade names can comprise mixtures of the various discrete mineral entities. Such mixtures of the smectite minerals are suitable for use in the invention.

While any of the smectite clays above are useful herein, certain clays are preferred. Smectite clays of a neutral or white colour are preferred when formulating white, or neutral granular compositions.

The "Brebent" and "Fulgel" clays which are particularly useful in this invention have a mean particle size between 5 and 25 µm, as determined by Malvern Mastersizer (Trade Mark) using the method described hereinafter. These clays contain relatively high levels of sodium, but are derived from calcium clays. Clays similar to this, which are suitable for the invention, have a sodium content less than about 4 per cent by weight, expressed as Na_2O and, typically, a calcium content less than 1.5 per cent by weight, expressed as CaO.

The granules prepared according to the invention comprise a water insoluble inorganic material. In this context, "water insoluble" is used to describe a compound with a solubility of less than 5 g per 100 g water at 25° C, preferably having a solubility less than 1 g per 100g water at 25° C. Suitable water insoluble inorganic materials include silica, aluminosilicates, aluminas, calcium carbonate, barium sulphate, titanium dioxide and pigments.

Preferred water insoluble inorganic materials are silica, materials consisting of at least 70 per cent silica by weight and aluminosilicates.

Preferably, the water insoluble inorganic materials have an average particle size below 10 µm and, more preferably, the average particle size is below 5 µm, as measured using a Malvern Mastersizer (Trade Mark) using the method described hereinafter.

When the granular material of the invention is used in detergent tablets, particularly useful insoluble inorganic materials are crystalline aluminosilicates, usually zeolites, particularly zeolites of use in a detergent formulation. Useful zeolites include zeolite P, A or X or mixtures thereof, with zeolite P being preferred.

In general, the empirical formula of a zeolite is

$$M_{2/n}O \cdot Al_2O_3 \cdot xSiO_2 \cdot yH_2O$$

wherein M represents a metallic cation having a valency of n, x indicates the ratio of atoms of silicon to atoms of aluminium and y indicates the ratio of molecules of water to atoms of aluminium. Many different types of zeolite, with varying ratios of silica to alumina, are known. Commonly, however, M is an alkali metal.

Zeolites of use in this invention may have the structure of any of the known zeolites. The structure and characteristics of many zeolites are described in the standard work "Zeolite Molecular Sieves" by Donald W. Breck, published by Robert E. Krieger Publishing Company. Usually, the value of x in the above empirical formula is in the range 1.5 to 10. The value of y, which represents the amount of water contained in the voids of the zeolite, can vary widely. In anhydrous material y = 0 and, in fully hydrated zeolites, y may be up to 5.

Zeolites useful in this invention may be based on naturally-occurring or synthetic aluminosilicates and the preferred forms of zeolite have the structure known as zeolite P, zeolite A or zeolite X. Particularly preferred forms of zeolite are those disclosed in EP-A-0 384 070, EP-A-0 565 364, EP-A-0 697 010, EP-A-0 742 780, WO-A-96/14270, WO-A-96/34828 and WO-A- 97/06102, the entire contents of which are incorporated herein by this reference. The zeolite P described in EP-A-0 384 070 has the empirical formula given above in which M represents an alkali metal and x has a value up to 2.66, preferably in the range 1.8 to 2.66, and has a structure which is particularly useful in the present invention.

It has been found that granular disintegrants of this invention are more effective when zeolites used to prepare them contain relatively little water. The preferred amount of water in a zeolite used to prepare the granules depends upon the type of zeolite. A particularly useful form of zeolite P contains less than about 16 per cent water by weight and, preferably, from about 9 to about 12 per cent water by weight.

The relative amounts of swelling clay and crystalline aluminosilicate in the granular disintegrant is preferably from 9 : 1 to 1 : 9 by weight clay : aluminosilicate and more preferably from 6 : 1 to 1 : 5 by weight clay : aluminosilicate.

Alkali metal aluminosilicates, especially zeolites, are commonly used in detergent compositions as a detergency builder. Where cleaning compositions prepared using the disintegrants of the invention contain an alkali metal aluminosilicate as detergency builder, at least part of the aluminosilicate constituent of the cleaning composition will be provided by the disintegrant granules.

When used, the water-swellable agent preferably has an average primary particle size up to about 600 μm, but, conveniently, has an average primary particle size of no more than 200 μm, preferably no more than 100 μm, and a water swelling capacity of at least 5 cm^3/g.

preferably at least 10 cm^3/g and more preferably at least 20 cm^3/g as determined in the test described hereinafter.

Typically the water-swellable agent comprises a polymer, frequently a wholly or partially cross-linked polymer, e.g. natural cellulose, cross-linked cellulose, (sodium) carboxymethyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross-linked starch, or cross-linked polyvinyl pyrrolidone. Currently preferred are Aquasorb A500 (ex Hercules), Ac-Di-Sol (ex FMC Corp) and Nilyn XL-90 (ex FMC Corp).

The granules of swelling clay and water insoluble inorganic material and, in the preferred embodiment, water-swellable agent are prepared by a dry granulation process. A typical known process for producing granules of inorganic material comprises mixing the inorganic material with a water or an aqueous liquid, agitating the wet mixture until granules are formed and subsequently removing water from the granules. In the dry granulation process of the current invention the granules are produced without the use of water or an aqueous liquid. The dry granulation process can be any of the processes that will be known to those skilled in the art, e.g. by blending the dry ingredients in a mixer followed by compacting on a roller compactor. Suitable mixers include high shear mixers such as a Pek mixer available from George Tweedy & Co of Preston - 28lb S.A. Machine, a Henschel mixer available from Thyssen Henschel Industrietechnik, Kassel, Germany and a Lodige mixer available from Gebr. Lodige Maschinebau, Paderborn, Germany, as well as low/medium shear mixers such as the orbiting screw Nautamixer available from Vrieco-Nauta, Holland or a ribbon mixer as supplied, for example, by Morton, Motherwell, Scotland. Suitable roller compactors include the Alexanderwerk WP50 manufactured by Alexanderwerk AG, Remscheid, Germany, the IR520 Chilsonator available from Fitzpatrick Company, Elmshurst, Illinois, USA and a roller compactor from Hosokawa Bepex of Minneapolis, USA.

A typical small-scale preparative method in which the product granule comprises swelling clay, crystalline aluminosilicate and a water-swellable agent is now described in detail. Swelling clay and aluminosilicate and water-swellable agent particulates are blended together, in the appropriate proportions, in a Pek mixer for 30 minutes. A minimum of 2 kg of blended material so prepared is compacted by feeding into an Alexanderwerk roller compactor, fitted with a sintered block vacuum deaeration system. The roller pressure setting is selected according to the strength of granule desired, higher pressures leading to stronger granules. Generally, roller pressure is between 8 and 25 MPa and a typical roller pressure is 10 MPa. The compacted material from the compactor is fed into a granulator, which forms part of the machine, and forced through a mesh and the resulting granules are then screened to the desired particle size range, e.g. an average particle size of 500 to 3000 µm, using standard laboratory sieves. Preferably, the particles have a size range of 700 to 1500 µm. The granules, however produced, comprise an intimate mixture of particles of swelling clay, crystalline aluminosilicate and water-swellable agent.

The moulded body which can be formed using the disintegrants produced according to the current invention can be a body useful in any application where a rapid disintegration is desired when the body is mixed with an aqueous liquid. Such applications include compositions intended to be used, for example, in agricultural sprays, in an industrial or process waste stream and in a body of water such as a lake or swimming pool. The moulded body may also comprise a foodstuff or a pharmaceutical, including a veterinary product. Particularly preferred moulded bodies are those used as cleaning compositions in home or personal care.

A moulded body formed from a cleaning composition using the disintegrants of the present invention may consist wholly of the cleaning composition or alternatively the moulded body may comprise a number of discrete portions, at least one of which comprises a cleaning composition containing a disintegrant prepared in accordance with the invention. In this event, the remaining portion or portions of the moulded body may be constituted by at least one other ingredient, usually one suitable for use in detergent and cleaning applications such as fabric washing, dishware washing, stain removal and water softening.

Cleaning compositions may also contain, in addition to the disintegrants of the invention, as essential ingredients, one or more detergency builders (wholly or partly incorporated in the disintegrant granules), and/or one or more detergent-active compounds which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof and/or other conventional additives.

Generally, cleaning compositions prepared using the disintegrants prepared according to the invention will contain from 1 to 20 per cent by weight of the disintegrant granules based on total weight of the composition. Frequently the cleaning compositions will contain from 4 to 10 per cent by weight of the disintegrant granules.

A suitable type of cleaning composition for use in most automatic fabric washing machines contains both anionic and nonionic surfactants. Cleaning compositions may also suitably contain a bleach system. Machine dishwashing compositions may suitably contain a chlorine bleach, while fabric washing compositions may contain peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. The skilled detergent worker will have no difficulty in applying the normal principles to choose a suitable bleach system.

Other materials that may be present in detergent compositions using the disintegrants prepared according to the invention include sodium silicate, fluoroscers, antiredeposition agents, inorganic salts such as sodium sulphate, enzymes, lather control agents or lather boosters as appropriate, pigments, and perfumes. This list is not intended to be exhaustive.

Procedures and Tests

Detergent Tablet Production

Detergent tablets used in the Examples that follow were produced using a 45 mm diameter die set (stainless steel) in conjunction with a Universal Testing Machine Type No. Z030 from Zwick GmbH, Ulm, Germany. A known quantity, 40-45 grams, of the cleaning composition which comprised the compositions mentioned in the Examples below was placed in the die, the die plunger was inserted and the assembly was placed between the platens of the Zwick machine which was operated to apply a predetermined pressure to produce a tablet having a defined density and, in particular, a dimensionally stable and fracture resistant tablet. Tablets thus produced were cylindrical in shape, with a diameter of about 45 mm and a height of about 20 mm. Tablets prepared had strength values in the range 25 to 32 kPa (as determined by the Diametrical Fracture Stress test described below) which are typical values for commercial fabric washing tablets found in the Western European market in 1999-2000.

Determination of Diametrical Fracture Stress

The method measures the strength of a detergent tablet using a Zwick Universal Testing Machine Type No. Z030 from Zwick GmbH, Ulm, Germany. The test determines the maximum force required to break the tablet in Newtons. The Diametrical Fracture Stress (DFS) is calculated using this resultant force and the dimensions of the tablet.

The exact dimensions of the tablet to be tested are measured using callipers and the tablet is placed between the platens of the Zwick Universal Testing Machine so that the machine will apply a force along the diameter of the tablet. The force required to break the tablet is then recorded using the following machine settings:

50 kN measuring head and platens.

Hard stops positioned at 375 mm and 1500 mm

LE position 48 mm (or adjusted appropriately if measured diameter of tablet differs from 45 mm).

Approach Speed of platens under test 10 mm/min.

The Diametrical Fracture Stress is calculated from this force using the equation

$$\text{Diametrical Fracture Stress (in Pa)} = \frac{2 \times \text{Force (in N)}}{\pi \times \text{Diameter (in m)} \times \text{Thickness (in m)}}$$

Determination of tablet disintegration profile

Method 1 (Dynamic)

The tablet disintegration profile provides an indication of the extent to which various tablets (e.g. different compositions, different densities) disintegrate under the defined conditions.

4500 g of demineralised water at 20° C were added to a 5 litre vessel fitted with pH, conductivity and temperature probes and maintained at a constant temperature of 20° C by immersion in a water bath. The tablets to be tested were inserted into a metal cage having the dimensions 9 cm x 4.7 cm x 2.7 cm and having 16 apertures (each about 2 mm square) per cm^2. The metal cage was attached to the shaft of an overhead stirrer (Heidolph/Janke and Kunkel stirrer) to allow it and its contents to be rotated while immersed in the demineralised water. Prior to testing, the empty cage was immersed in the demineralised water and rotated at 80 rpm for a short period of time until the temperature of the demineralised water as detected by the temperature probe had stabilised at 20 ± 0.2° C. At this time, the conductivity, pH and temperature values registered by the respective probes were recorded. The stirrer was then switched off to allow the cage to be raised out of the water so that a pre-weighed detergent tablet to be investigated could be inserted into the cage. The cage was then re-immersed in the demineralised water together with the inserted tablet and the stirrer was switched on to resume rotation of the container at 80 rpm. Measurements of conductivity and pH, initially at 15 second intervals for one minute and thereafter at one minute intervals, were made over a period of 10 minutes after which time the cage was raised out of the demineralised water to allow the residue of the tablet to be removed. The residue was then dried in an oven at 105° C so that the dry weight of the residue could be calculated as a percentage of the original tablet weight. This procedure was repeated for a number of tablets having different compositions and different densities.

Method 2 (Static)

4500 g of tap water at 20° C were added to a 5 litre vessel, which was maintained at 20° C by immersion in a water bath. The tablets to be tested were weighed and inserted into a metal cage 20 cm in diameter with 1 cm^2 perforations. The cage was lowered into the 5 litre vessel and left for 60 seconds. The cage was then removed from the water, residue of the tablets was placed on an aluminium tray, dried for 24 hrs at 105° C and weighed to determine the percentage which had not disintegrated.

Determination of water swelling capacity of water-swellable agent

To demonstrate the water swelling capacity of the water-swellable agent, 19.6 g of the agent was blended with 0.4 g of ultramarine pigment and compressed into a tablet using a laboratory tablet press at about 250 MPa to give a tablet 32 mm in diameter. This was crushed and sieved to give granules 500-1000 µm in size. A glass tube, 33 mm in internal diameter and

about 30 cm long with a sintered porous glass disc (porosity 1) fitted at one end was immersed upright, with said one end lowermost, in a large beaker of water (at 25° C) so that the water level rose to about 14 cm above the sintered glass. 1 g of granules was added to the tube and allowed to settle onto the sintered glass disc. With this arrangement water has access to the granules from both above and below. The granules immediately began to swell, forming a jelly-like mass. The ultramarine pigment imparted a blue colour to the mass making it easy to see the top and to record its height. The height of the swelling mass was recorded at intervals and showed an initial rapid rise followed by a levelling off after about 20-30 minutes. From the diameter of the tube, the volume of the swollen mass can be calculated. The result was expressed as cm^3/g water-swellable agent after 20 minutes.

Determination of Weight Mean Particle Size

The weight mean particle size of the materials used in this invention is determined using a Malvern Mastersizer model X, with a lens range up to 300 mm RF and MS17 sample presentation unit. This instrument, made by Malvern Instruments, Malvern, Worcestershire, uses the principle of Mie scattering, utilising a low power He/Ne laser. Before measurement the sample is dispersed ultrasonically in water for 7 minutes to form an aqueous suspension. This suspension is stirred before it is subjected to the measurement procedure outlined in the instruction manual for the instrument, utilising the 300 mm RF lens range in the detector system. The Malvern Mastersizer measures the weight particle size distribution of the inorganic material or reference material. The weight mean particle size (d_{50}) or 50 percentile is readily obtained from the data generated by the instrument.

The invention is illustrated by the following non-limiting examples.

EXAMPLES

Example 1

Disintegration profiles were investigated for a number of detergent tablet formulations, all based on a standard detergent base powder having the following composition. This composition is representative of a typical composition used in a European detergent formulation for tablet detergents, but without minor additives, such as perfume.

Ingredient	% by weight
Sodium linear alkylbenzene sulphonate	12.34
Nonionic surfactant	5.42
Zeolite[1] (anhydrous)	27.60
Sodium acetate trihydrate/zeolite blend (99 : 1 blend)	3.53
Sodium carbonate	4.10
Sodium citrate dihydrate	5.07
TAED granules (83% active)	5.44
Sodium percarbonate (coated)[2]	20.30
Sodium silicate granules (80% active)	4.35
Minor ingredients (fluorescer, polymer, sequestrants, etc.)	6.88
Salts, moisture and non-detergent organic matter	4.97
	100.00

[1] Doucil A24, a maximum aluminium P zeolite available from Crosfield Ltd. UK

[2] Coated percarbonate available from Interox.

The detergent powder was blended with 10 per cent by weight amounts of disintegrant granules having different compositions. In each instance, the disintegrant granules comprised a water insoluble inorganic material, in the form of a zeolite, a swelling clay and a water-swellable agent. In the Examples given below, Doucil A24 (Trade Mark) is a P-type zeolite sold by Crosfield Limited of Warrington, UK; Doucil 4A (Trade Mark) is a 4A zeolite obtainable from Crosfield Limited; Brebent is a sodium enriched calcium-form bentonite available from Laporte plc and the water-swellable agent was Ac-Di-Sol, a croscarmellulose sodium available from FMC Corporation, Philadelphia USA.

The different formulations of the disintegrant granules (on a by weight basis) are given in Table 1. For comparison, a granule (Granule F) was prepared using only a zeolite and a swelling polymer. All these granules were prepared using a Pek mixer and Alexanderwerk roller compactor as hereinbefore described. Each was classified by sieving to a size range of 500 to 1200 µm.

TABLE 1

Granule Code	Wt. % of Ac-Di-Sol in granule	Wt % of Doucil A24 in granule	Wt % of Brebent in granule
A	7.5	20.10	72.40
B	7.5	33.20	59.30
C	7.5	46.25	46.25
D	7.5	59.30	33.20
E	7.5	72.40	20.10
F (comparative)	7.5	92.50	0

A series of tablets were prepared at a pressure shown in Table 2 below. Tablets 1A to 1E contain 10% by weight of granules A to E respectively, the other 90% being formed from the base detergent powder mentioned above.

Table 2 shows the level of disintegration after immersion in water for these tablets using the experimental protocol described above in Method 2. The measurement of disintegration shows the level of disintegration obtained by subtracting the percentage of undisintegrated residue retained in the 'cage' from 100.

TABLE 2

Tablet Code	Pressure of formation ($N\ mm^{-1}$)	Disintegration % on storage			
		0 days	7 days	14 days	28 days
1A	4.50	80	87	93	100
1B	4.50	75	91	93	96
1C	4.50	71	100	96	100
1D	4.50	54	79	85	79
1E	4.25	41	61	67	75
1F	5.00	81	58	48	42

Example 2

Detergent tablets were prepared in a similar manner to that described in Example 1 using granules having the compositions given in Table 3. The granules were classified to a size range of 500 to 1200 μm.

TABLE 3

Granule code	Wt % Ac-Di-Sol in Granule	Wt % Doucil A24 in granule	Wt % Brebent in granule
G	7.50	59.30	33.20
H	7.50	46.25	46.25
I	7.50	33.20	59.30

A series of tablets were prepared at a pressure shown in Table 4 below . Tablets 2G to 2I contain 5% by weight of granules F to I respectively, the other 95% being formed from a mixture of the base detergent powder mentioned above and sodium acetate (15% by weight of the tablet). The sodium acetate used was 99% by weight sodium acetate trihydrate and 1% by weight zeolite, Doucil A24.

Table 4 shows the level of disintegration after immersion in water for these tablets using the experimental protocol described above in Method 2. The measurement of disintegration shows the level of disintegration obtained by subtracting the percentage of undisintegrated residue retained in the 'cage' from 100.

TABLE 4

Tablet Code	Pressure of formation ($N\ mm^{-1}$)	Disintegration % on storage				
		0 days	2 days	7 days	14 days	28 days
2G	4.30	97	89	82	88	87
2H	4.25	92	90	90	89	89
2I	4.30	92	96	86	83	81

Example 3

Tablets were prepared according to the method of Example 2 using Granule C and Granule F. Tablet 3C contained 15% by weight sodium acetate (as in Example 2) and Tablet 3F contained 20% sodium acetate. The disintegration results are shown in Table 5 below.

TABLE 5

Tablet Code	Pressure of formation (N mm^{-1})	Disintegration % on storage				
		0 days	2 days	7 days	14 days	28 days
3C	4.30	100	95	80	NA	NA
3F (comparative)	4.25	92	90	90	89	89

NA = Not available

Example 4

Granules were prepared using a Pek mixer and Alexanderwerk roller compactor as hereinbefore described. Each was classified by sieving to a size range of 500 to 1200 μm. Granule J was prepared using Doucil A24 and Granule K was prepared using Doucil 4A, a zeolite A available from Crosfield Ltd., Warrington, UK. Each contained 50% by weight zeolite and 50% by weight Brebent clay.

Tablets were prepared according to the method of Example 2 using these granules and containing 10% by weight sodium acetate (as in Example 2). The disintegration results are shown in Table 6 below.

TABLE 6

Tablet Code	Pressure of formation (N mm^{-1})	Disintegration % on storage			
		0 days	7 days	14 days	28 days
4J	3.7	57	63	61	72
4K	3.7	33	47	53	63

<u>Example 5</u>

Example 4 was repeated except that the granules were incorporated in the tablets at 5% by weight and the amount of sodium acetate (as in Example 2) used in the tablets was 15% by weight. The disintegration results are shown in Table 7 below.

TABLE 7

Tablet Code	Pressure of formation (N mm^{-1})	Disintegration % on storage				
		0 days	2 days	7 days	14 days	28 days
5J	4.2	83	81	82	76	76
5K	4.2	79	67	64	63	63

CLAIMS

1. A process for the preparation of a disintegrant, suitable for use in a composition in the form of a moulded body, comprising forming by a dry granulation process a granular composition comprising a swelling clay and a water insoluble inorganic material.

2 A process for the preparation of a disintegrant, suitable for use in a composition in the form of a moulded body, comprising forming by a dry granulation process a granular composition comprising a swelling clay, a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent of the combined weight of said swelling clay, said water insoluble material and said water-swellable agent.

3. A process according to claim 1 or 2 characterised in that the dry granulation process comprises blending ingredients of the granular composition in a mixer followed by roller compaction of the mixture so produced.

4. A process according to any one of the preceding claims characterised in that the roller pressure during roller compacting is in the range 8 to 25 MPa.

5. A process according to any one of the preceding claims characterised in that the granules are screened to a size in the range 500 to 3000 μm.

6. A composition suitable for use as a disintegrant in a composition in the form of a moulded body, said composition being in the form of granules comprising a swelling clay, a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent of the combined weight of said swelling clay, said water insoluble inorganic material and said water-swellable agent.

7. A composition according to claim 6 characterised in that the water-swellable agent is present in an amount comprising no more than 7.5 per cent of the combined weight of said swelling clay, said water insoluble inorganic material and said water-swellable agent.

8. A composition according to claim 6 or 7 characterised in that the water-swellable agent is present in an amount comprising at least 1 per cent of the combined weight of said swelling clay, said water insoluble material and said water-swellable agent.

9. A composition according to any one of claims 6 to 8 characterised in that the swelling clay is a smectite clay.

10. A composition according to claim 9 characterised in that the smectite clay is a bentonite clay.

11. A composition according to any one of claims 6 to 10 characterised in that the water insoluble inorganic material is silica, a material containing at least 70 per cent silica by weight or an aluminosilicate.

12. A composition according to claim 11 characterised in that the water insoluble inorganic material is a crystalline aluminosilicate which is a zeolite having the empirical formula

$$M_{2/n}O \cdot Al_2O_3 \cdot xSiO_2 \cdot yH_2O$$

wherein M represents a metallic cation having a valency of n, x indicates the ratio of atoms of silica to atoms of aluminium and y indicates the ratio of molecules of water to atoms of aluminium.

13. A composition according to claim 12 characterised in that the zeolite is a zeolite P, a zeolite A or a zeolite X.

14. A composition according to claim 12 or 13 characterised in that the zeolite is a zeolite P in which M is an alkali metal and x has a value in the range 1.8 to 2.66.

15. A composition according to claim 12, 13 or 14 characterised in that zeolite is a zeolite P having a water content in the range 9 to 12 per cent by weight of the zeolite.

16. A composition according to any one of claims 6 to 15 characterised in that the relative amounts of swelling clay and crystalline aluminosilicate in the granular disintegrant are in the ratio of 9 : 1 to 1 : 9 by weight clay : aluminosilicate.

17. A composition according to any one of claims 6 to 16 characterised in that the water-swellable agent has an average primary particle size of up to 600 µm.

18. A composition according to any one of claims 6 to 17 characterised in that the water-swellable agent has a water-swelling capacity of at least 5 cm^3/gram.

19. A composition according to any one of claims 6 to 18 characterised in that the water-swellable agent is natural cellulose, cross-linked cellulose, carboxymethyl cellulose, sodium carboxymethyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross linked starch, or cross linked polyvinyl pyrrolidone.

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ABSTRACT

A process for the preparation of a disintegrant, suitable for use in a composition in the form of a moulded body, comprises forming by a dry granulation process a granular composition comprising a swelling clay and a water insoluble inorganic material. Preferably, the granular composition also comprises a water-swellable material. The invention also makes available a composition suitable for use as a disintegrant in a composition in the form of a moulded body, said composition being in the form of granules comprising a swelling clay, a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent of the combined weight of said swelling clay, said water insoluble inorganic material and said water-swellable agent.